भारतीय मानक
Indian Standard

IS 1448 [P : 133] : 2018

पेट्रोलियम और उसके उत्पादों के लिए परीक्षण के तरीके

[पी : 133]

कैलीसींड पेट्रोलियम कोक का असली घनत्व का निर्धारण

(पहला पुनरीक्षण)

Methods of Test for Petroleum and Its Products

[P : 133]

Determination of Real Density of Calcined Petroleum Coke

(First Revision)

ICS 75.100

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March 2018 **Price Group 1**

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard [P : 133] (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Real density is the mass divided by the volume occupied by the material excluding pores and voids. It is required, therefore, that voids in the coke be eliminated and that pores in the material be filled by the fluid being displaced. The real density of calcined petroleum coke directly influences the physical and chemical properties of the manufactured carbon and graphite artifacts for which it is used.

This standard was initially published in 1989. In this revision reporting has been included and precision is modified.

In reporting the results of a test or analysis made in accordance with this standard, if the final values, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 133]

DETERMINATION OF REAL DENSITY OF CALCINED PETROLEUM COKE

(First Revision)

1 SCOPE

This standard describes an analytical test procedure for the determination of real density of calcined petroleum coke (CPC).

2 APPARATUS

2.1 Analytical Balance — Accurate to 0.1 mg.

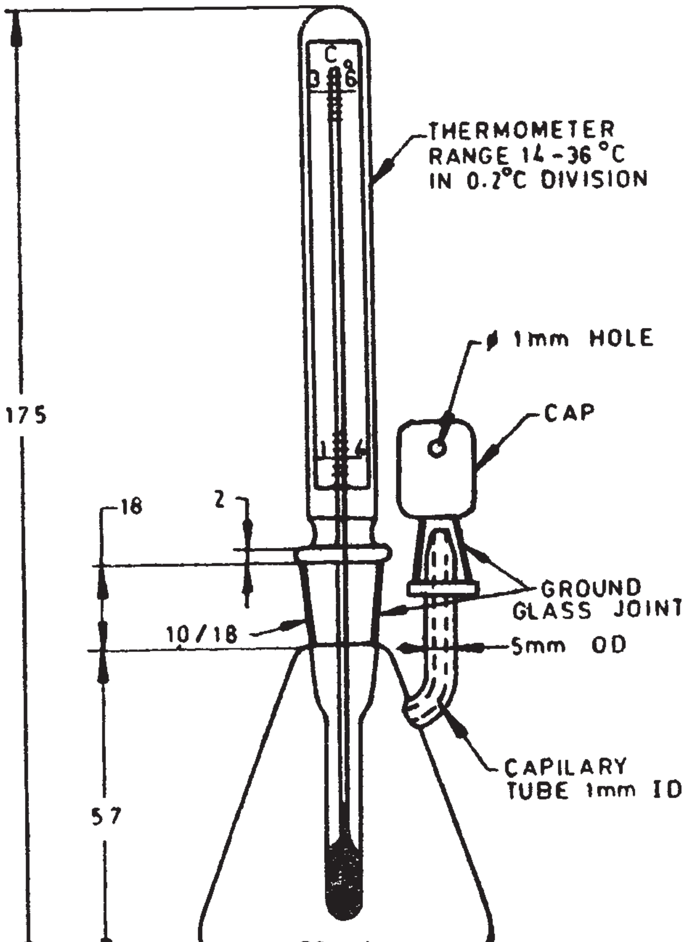
2.2 Specific Gravity Bottle (Pyknometer) — 50-ml capacity with ground joint thermometer, range 14 to 36°C graduated in 0.2°C and capillary side tube with cap (*see* Fig. 1).

2.3 Water Bath — Stainless steel with stirrer.

2.4 Hot Plate

2.5 Ball Mill — Two tiers, two jar capacity or any other suitable milling equipment capable of grinding 75 micron particles.

2.6 Ball Mill Jar — Approximately 1.4 litre capacity,



All dimensions in millimetres.

FIG. 1 SPECIFIC GRAVITY BOTTLE (PYKNOMETER) 50 ml CAPACITY WITH INTERCHANGEABLE THERMOMETER

made of highest quality white vitrified porcelain fortified with alumina for extra density and resistance to abrasion and impact.

2.7 Flint Pebbles — About 16 mm dia (grinding media).

3 PROCEDURE

3.1 Calibration of Pyknometer

Weigh an empty clean and dried pyknometer, complete with the thermometer and cap, to the nearest 0.2 mg; designate this mass as 'M'. After the pyknometer has been weighed, remove the thermometer and cap.

3.1.1 Fill the pyknometer with freshly boiled distilled water (boiled for 30 min) up to the neck. Cool to below 25°C by immersing the pyknometer in ice cold water up to the neck and adjust the water level to about half way in the thermometer socket. Insert the thermometer and allow the excess water to flow out through the side arm. Do not place the capillary cap on at this time. Place the filled pyknometer in the water bath adjusted to 25 ± 0.05°C.

3.1.2 Allow the pyknometer to remain in the bath until the thermometer reads 25°C. Then, while still in the bath, adjust level of water in the capillary arm until the level is even with the top of the ground glass section of the arm. Using a piece of adsorbent paper, completely wipe the outside of the ground glass arm dry, place the cap into its position on the arm and remove the pyknometer from the bath. Wipe the assembled pyknometer completely dry.

3.1.3 Remove any electrostatic charge on the pyknometer generated by wiping, by placing the pyknometer in an electrically grounded wirebasket for about 30 s. Remove the pyknometer and quickly weigh it to the nearest 0.2 mg. Designate this mass as M_2

3.1.3.1 Calculate the mass of water content as:

$$= M_2 - M \text{ at } 25\ ^\circ\text{C}$$

3.1.4 This procedure should be repeated several times and an average taken to ensure correct calibration of the pyknometer.

3.2 Determination of Real Density

3.2.1 Weigh the clean and dry pyknometer (calibrated earlier) alongwith thermometer and cap. Let the mass be 'M' (nearest to 0.2 mg). Remove the thermometer. Weigh approximately 5 g of dried coke samples passing through 75 micron sieve and transfer into the bottle with the help of a funnel, taking care not to get any of the samples on the ground glass socket. Replace the thermometer and reweigh the pyknometer plus sample. Designate this mass as M_1.

3.2.2 Take off cap and thermometer and fill the bottle to half with distilled water. Shake gentlyto help coke and water to mix. Place the bottle on the hot plate and boil contents for 1 h.To keep bottle half-full and to wash down any coke adhering to the sides, add small amounts of hot water from time-to-time. Shaking occasionally also helps to keep the coke from adhering to the sides. After 1 h, remove it from hot plate.

3.2.3 Fill the pyknometer with freshly boiled distilled water (boiled for 30 min) up to the neck. Cool to below 25°C by immersing the pyknometer in ice cold water up to the neck and adjust the water level to about half way in the thermometer socket. Insert the thermometer and allow the excess of water to flow out through the side arm. Do not place the capillary cap on at this time. Place the filled pyknometerin the water bath adjusted to 25 ± 0.05°C.

3.2.4 Allow the pyknometer to remain in the bath until the thermometer reads 25°C. Then while still in the bath adjust level of water in the capillary arm until the level is even with the top of the ground glass section of the arm. Using a piece of adsorbent paper, completely wipe the outside of this ground glass arm dry, place the cap into its position on the arm and remove the pyknometer from the bath. Wipe the assembled pyknometer completely dry.

3.2.5 Remove any electrostatic charge on the pyknometer generated by the wiping, by placing the pyknometer in an electrically grounded wire basket for about 30 s. Remove the pyknometer and quickly weigh it to the nearest 0.2 mg. Designate this mass as M_3.

4 CALCULATION

$$\text{Real density (g/ml)} = \frac{M_1 - M}{(M_2 + C) - M_3} \times D_w$$

where

M = mass in g of empty pyknometer;
M_1 = mass in g of pyknometer and coke sample;
M_2 = mass in g of pyknometer filled with water at 25°C;
M_3 = mass in g of pyknometer, coke sample and water;
C = mass in g of coke sample = $M_1 - M$; and
D_W = true density of water at 25°C = 0.997 07

5 REPORTING

Report the apparent densities to two decimal places.

6 PRECISION

Method precision shall be established based on round robin outcome.

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This Indian Standard has been developed from Doc No.: PCD 01 (2881).

Amendments Issued Since Publication

Amendment No.	Date of Issue	Text Affected

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Published by BIS, New Delhi